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(54) PRODUCTION OF EMULSIFIED FAT AND OIL COMPOSITION

(57) Abstract:

PURPOSE: To obtain an emulsified fat and oil composition having uniform particle diameter, free from the separation between water phase and oil phase or the sanitary problem due to the distribution of particle diameter and having excellent physical property, feeling of food and taste.

CONSTITUTION: The emulsified fat and oil composition having average particle diameter of 1-20 times as large as the pore diameter of a porous membrane having uniform pore diameter is previously prepared and the emulsified fat and oil composition is remulsified by passing through the porous membrane having uniform pore diameter.

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A METHOD FOR THE MANUFACTURE OF EMULSIFIED FAT/OIL COMPOSITION

Abstract:

[Objects]

To offer an emulsified fat/oil composition where the particle size is uniform, there is no separation of aqueous and oily phases due to particle size distribution, there is no hygienic problem and physical property, feel on eating and taste which have not been available are achieved.

[Constitution]

An emulsified fat/oil composition having an average particle size which is about 1- to 20-fold of the pore size of a porous membrane having a uniform pore size is previously prepared and then the said emulsified fat/oil composition is passed through the above-mentioned porous membrane having a uniform pore size to re-emulsify.

What is Claimed is:

1. A method for the manufacture of an emulsified fat/oil composition, characterized in that, an emulsified fat/oil composition having an average particle size which is about 1- to 20-fold of the pore size of a porous membrane having a uniform pore size is previously prepared and then the said emulsified fat/oil composition is passed through the above-mentioned porous membrane having a uniform pore size to re-emulsify.

2. The method for the manufacture of an emulsified fat/oil composition according to claim 1, wherein an average particle size of the emulsified fat/oil composition obtained by passing through the porous membrane is from 1- to 3-fold of the pore size of the porous membrane.

3. The method for the manufacture of an emulsified fat/oil composition according to claim 1, wherein an emulsified fat/oil composition of a water-in-oil type where inner phase is water while outer phase is oil is previously prepared and then passed through the porous membrane.

4. The method for the manufacture of an emulsified fat/oil composition according to claim 1, wherein an emulsified fat/oil composition of an oil-in-water type where inner phase is oil while outer phase is water is previously prepared and then passed through the porous membrane.

5. The method for the manufacture of an emulsified fat/oil composition according to claim 3 or 4, wherein an HLB of an emulsifier which is added to the aqueous phase of the previously-prepared emulsified fat/oil composition is 10.0 or more.

Detailed Description of the Invention:

[Technical Field of the Invention]

The present invention relates to a novel method for the manufacture of an emulsified fat/oil composition such as an emulsified fat/oil composition of a water-in-oil type (hereinafter, referred to as "W/O type emulsion") or an emulsified fat/oil composition of an oil-in-water type (hereinafter, referred to as "O/W type emulsion") and, more particularly, it relates to a method for the manufacture of an emulsion having a narrow particle size distribution and

calorie low due to a recent tendency to health food in recent dietary life and, in that case, an emulsion of very fine particles is prepared using a high-pressure homogenizer or the like so that a large amount of water is entrapped therein. However, in an emulsion where particles are made very fine by a high-pressure homogenizer or the like, although its particle size distribution is very narrow, the particle size is just made small and control to a uniform particle size is difficult. In addition, as a result of an increase in a volume percentage, viscosity increases suddenly whereby the operation during the manufacturing process becomes difficult. The present inventors have analyzed the stirrer or the homogenizer which is usually used in the manufacture of emulsions and have confirmed that mere shearing stress given as such results in a wide particle size distribution and an insufficient stability. Even when polyglycerol fatty acid ester which is an emulsifier designed to give a strong emulsifying stability is used, although the stability is improved and distribution of emulsion particles becomes somewhat uniform, a sudden increase in viscosity of emulsion due to an increase in the volume percentage of the inner phase and also generation of smell of emulsifier are confirmed.

As such, it is important for both W/O and O/W type emulsions to control the particle size of the emulsion for giving various functions but, in the conventional methods where an emulsifier is added or a shearing stress is applied, the resulting particle size distribution has some width and it is difficult that an aimed particle size is stably and uniformly controlled. However, the significance and request for an easy control of particle size of emulsion are very big.

[Problems to be Solved by the Invention]

In order to overcome such a problem, utilization of a membrane has been investigated recently as an application to emulsifying technique of emulsions. Research, development and application of utilization of membrane were already carried out briskly in the areas of filtration, concentration and filtration while its application to food has been started only recently and a method for the manufacture of an emulsion using a porous glass membrane (Japanese Laid-Open Patent Publication 02/095,433) is one of its examples. Unlike a homogenizer, this porous glass membrane does not require high pressure and a very homogeneous emulsion can be prepared by that easily whereby the said method is an epoch-making emulsifying method.

However, in an application to emulsified fat/oil compositions for processed food represented by margarine (O/W type) and whipped cream (W/O type), an emulsion having poor emulsifying stability is afforded because of entirely different wetting degree of membrane, resistance of membrane, viscosity, etc. depending upon the type of emulsifier, oil or additives whereby it sometimes happens that a dispersed phase (inner phase) is not well compressed into a continuous phase (outer phase). For example, in the case of a very sticky emulsifier such as lecithin, the emulsifier itself adheres to the wall surface of the porous membrane resulting in clogging whereby the liquid does not pass therethrough at all. As a result thereof, type of the emulsifier applicable is limited. In the case of a system where the outer phase is water such as an O/W type emulsion, it is necessary to add an ionic emulsifier or an additive to the side of water due to an electric property of the porous membrane *per se*. Accordingly, when main and auxiliary materials which have been commonly used for conventional emulsified fat/oil compositions are used, only a very limited range of utilization is possible and they are unable to be so much used for the above-mentioned emulsified fat/oil compositions for food processing.

[Means for Solving the Problems]

Under such circumstances, the present inventors have carried out an intensive investigation for a relationship between the emulsion particles and the emulsifying method for W/O and O/W type emulsions from a viewpoint of physical chemistry and chemical engineering with an expectation that the advantages of the above-mentioned porous membrane might be applied to emulsions for food processing and have found that, when a porous membrane is further applied to the already and preliminarily emulsified emulsion as a method for the preparation of emulsions, it is possible that an emulsion having a very uniform distribution can be controlled to any particle size whereupon the present invention has been achieved.

Thus, as a result of various studies on emulsifying stability, physical property, feel on eating and taste of the emulsion and also on type and adding amount of the emulsifier, the present inventors have found that size, distribution width, numbers and viscosity of the emulsion particles and also viscosity and surface tension of the inner and outer phases are big factors. The present invention has been achieved on the basis of such a finding.

In the present invention, a W/O type emulsion or an O/W type emulsion is

preliminarily emulsified using a commonly used stirring type emulsifier such as a chemistirrer or a homomixer so as to make its average particle size from 1- to 20-fold of the pore size of the porous membrane and then it is passed through a porous membrane to re-emulsify whereupon an emulsion having a uniform particle size is manufactured whereby it is possible to give an emulsion in which physical property, feel on eating and taste can be freely controlled and stability of emulsion is good.

The term "average particle size" of the emulsion is a value of an average particle size calculated from the particle sizes on a photographic taken for the emulsion particles under a microscope. The "re-emulsification" utilizing a porous membrane in the present invention means that emulsion having bigger size than the pore size of the porous membrane is finely divided in the said porous membrane and, therefore, it is an important point to prepare an emulsion having somewhat bigger size than the pore size of the porous membrane. In the present invention, an emulsion which is emulsified by an emulsifier such as a chemistirrer and having a particle size of from 1- to 20-fold of the pore size of the porous membrane as measured by the above measuring method is passed through the said porous membrane whereby it is possible to give an emulsion having a uniform distribution width.

With regard to the state of emulsion particles when passing through the porous membrane in the above-mentioned case, shape of the emulsion having bigger size than the pore diameter of the porous membrane changes from spherical to elliptic or slender shape. Accordingly, it is necessary to conduct a smooth fine dividing of emulsion and a formation of surface from such a deformed state. They are determined by the surface tension between porous membrane and the outer phase of the emulsion and, during the passing stage therethrough, they are determined by the relation between the fluidity characteristics and shearing stress received by the emulsion. Adjustment of the appropriate surface tension can be selected by investigating the function of the emulsifier. With regard to fluidity characteristics, the bigger the average particle size of the emulsion, the broader the particle size distribution width and the more the non-Newtonian property in the fluidity characteristics. Accordingly, the changing rate of the fluidity characteristics to the shearing stress is big as well and the influence by the shearing stress becomes large. Therefore, with regard to the stability of the emulsion particles when passing through the porous membrane, it is necessary to consider in the relation between pore diameter of the porous membrane and shearing stress/fluidity characteristics. From

}: no detailed analysis required

such a viewpoint, an emulsion having particles which are 20-fold or more of the pore size of the porous membrane has a big changing rate in fluidity characteristics to shearing stress and, accordingly, they are unable to pass through the porous membrane continuously against the delicate changes in shearing stress. In the case of emulsion having an average particle size of 20-fold or more, movement of the particles when passing through the porous membrane under the applied shearing stress is quick and more complicated whereby deformation of the particles is too vigorous resulting in destroy of the emulsion rather than formation of surface membrane whereupon a uniform emulsion is not available or, moreover, an emulsion where oily and aqueous phases are separated is resulted. However, in an emulsion of the present invention having an average particle size of 1- to 20-fold of the pore size of the porous membrane, it is possible to pass through the porous membrane constantly against the delicate change in shearing stress and, therefore, smooth fine dividing of emulsion takes place whereby it is easy to pass the emulsion through the porous membrane and the resulting emulsion has a uniform distribution.

There is no particular limitation for the fat/oil used in the present invention but any of animal/vegetable oil, hydrogenated animal/vegetable oil, fractionated oil, umuesu oil, etc. which has been conventionally used for the manufacture of emulsions may be appropriately used. Depending upon the tackiness of the emulsion, those fat/oil may be hydrogenated, fractionated or compounded.

There is no particular limitation for the emulsifier to be added to an oily phase but commonly used glycerol fatty acid ester, sucrose fatty acid ester, sorbitan fatty acid ester, propylene glycol fatty acid ester, polyglycerol fatty acid ester, polyglycerol condensed ricinoleate, lecithin, etc. may be used either solely or jointly by mixing two or more. There is no particular limitation for the adding amount of such an emulsifier so far as it is 0.1% or more. When, however, concentration of the emulsifier is too low, separation of the emulsion takes place quickly resulting in a problem in stability.

With regard to an emulsifier to be added to an aqueous phase, one or more of sucrose fatty acid ester, sorbitan fatty acid ester, polyglycerol fatty acid ester, lecithin, etc. having an HLB (hydrophobic/lipophilic balance) of 8.0 or more or, preferably, 10.0 or more may be used. Again, there is no particular limitation for the adding amount so far as it is 0.1% or more. When, however, concentration of the emulsifier is too low, separation of the

emulsion takes place quickly resulting in a problem in stability.

There is no particular limitation for the ratio of water to oil in a W/O type emulsion and that of oil to water in an O/W type emulsion but, in order to give a good emulsified state, the ratio of water to oil and that of oil to water is to be 5/5 or more or, preferably, 4/6 or more in both cases of W/O and O/W type emulsions.

Now, specific methods for the manufacture of an emulsified fat/oil composition according to the present invention will be as follows. First, in the case of a W/O type emulsion, an oily phase to which emulsifier, perfume, etc. are added is placed in a container, set with a stirrer such as a homo-mixer and heated to dissolve at 60-70°C with stirring. After that, a predetermined amount of aqueous phase part is gradually added thereto and the mixture is stirred at 60-70°C with stirring for 10 minutes or more to conduct a preliminary emulsification. Degree of stirring at this preliminary emulsification is made to such an extent that the particle size of the resulting emulsion is 1- to 20-fold of the pore size of the porous membrane used for the re-emulsification.

Then, deaeration is carried out to remove the air which was incorporated in the emulsion at the above preliminary emulsification. The deaeration operation may be carried out either by allowing to stand or *in vacuo*. Time for deaeration will be sufficient if it is ten minutes.

After such operations, the preliminarily emulsified emulsion is adjusted to the aimed temperature. Such a temperature is the temperature of the emulsion product which is a final product and can be appropriately selected depending upon the object.

After that, the preliminarily emulsified and deaerated emulsion is quantitatively passed through a porous membrane which was previously wetted with oil when the outer phase is an oily phase (W/O type) or with water when the outer phase is an aqueous phase (O/W type) using a gas such as nitrogen gas or/and a pump. The porous membrane used here is preferably in a tube or a plate when stability of the emulsion or operation, etc. are taken into consideration although the membrane is not limited thereto provided that the porous membrane has the same effect. It is difficult to grasp the passing rate of the emulsion through the porous membrane in detail but, in the present invention, "treating amount per

hour" is defined as an average passing rate. When this average passing rate is from 100 cc/minute to 5,000 cc/minute, an emulsion having a desired particle size can be prepared. In order to improve the stability of the emulsion in that case, it is preferred that the average passing rate is increased so that the passing pressure is made high. Incidentally, the particle size of the resulting emulsion is related to the pore size of the porous membrane and is controlled to an average particle size of from 1- to 3-fold of the said pore size. Accordingly, in order to give an emulsion having a small average particle size, it is preferred to select a porous membrane having a small pore size whereby the average passing rate is made slow. Incidentally, in the earlier-filed application (Japanese Patent Application Filing No. 095,433/90), it is mentioned that, from the relation between the porous membrane and the outer phase, particle size of the prepared emulsion is theoretically around 3.25-fold of the pore size of the porous membrane. Thus, in a method described in the earlier-filed application, it is necessary to select a porous membrane having a pore size of 0.3 micron or less in order to prepare an emulsion having an average particle size of 1 micron. Actually however, preparation of a porous membrane having a pore size of 0.3 micron or smaller is very difficult (in the porous glass membrane used in the earlier-filed application, it is described that 0.3 micron is the lowest limit) whereby it is not possible to prepare an emulsion having a particle size of 1 micron or smaller using the above-mentioned method. On the contrary, in a method using re-emulsification according to the present invention, particle size of the resulting emulsion is only 2- to 2.5-fold of the pore size of the porous membrane whereby, against the aimed particle size of the emulsion, the range of the pore size of the porous membrane is broader.

[Examples]

The present invention will now be further illustrated by way of the following examples and comparative examples although the present invention is not limited thereto. Incidentally, the terms "part(s)" and "%" used in the examples and the comparative examples are those by weight.

Evaluating items and methods for the evaluation will be as follows.

Particle size distribution: A few drops of a sample were placed on a sample stand (5.0 x 5.0 x 0.15 mm) on a slide and the particles were taken under a differential interference microscope (Olympus BH-2) with a magnification of 200-400. Particle sizes were measured from the picture taken using a

digitizer (KW4300; manufactured by Graphtec Corp.) and particle size distribution and average particle size (D_{av}) were calculated.

Stability of emulsion: An emulsion was taken in a 200-cc graduated cylinder and allowed to stand in an atmosphere of 20°C or 30°C and the separated amount (cc) of oil or water was measured.

Example 1 (O/W type emulsion)

(Preparation of Oily Phase) Corn oil (50 parts) was heated at 60°C and, under stirring with a chemistirrer (type B-100; manufactured by Tokyo Rikakikai Co.) at 620 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (50 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol condensed fatty acid ester of HLB = 10 (SY Glyster TS-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a chemistirrer at 620 rpm for 20 minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate.

(Preparation of O/W Type Emulsion) The preliminarily emulsified O/W type emulsion prepared as above was passed through a porous membrane (inner diameter of 9.0 mm; outer diameter of 10.0 mm, length of 100.0 mm, average pore size [dp] of 2.70 microns) at an average passing rate of 350 cc/ten minutes to re-emulsify whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 2.

Comparative Example 1 (O/W Type Emulsion)

(Preparation of Oily Phase) Corn oil (50 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (50 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol condensed fatty acid ester of HLB = 10 (SY Glyster TS-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a chemistirrer at 620 rpm for 20 minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 2.

Example 2 (W/O Type Emulsion)

(Preparation of Oily Phase) Soybean oil (50 parts) was heated at 60°C and, under stirring with a homo-mixer (TK Auto-Homomixer; manufacrured by Tokushu Kika Kogyo) at 3,000 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (50 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by stirring with a homo-mixer at 3,000 rpm for ten minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate.

(Preparation of W/O Type Emulsion) The preliminarily emulsified W/O type emulsion prepared as above was passed through a porous membrane (inner diameter of 9.0 mm; outer diameter of 10.0 mm, length of 100.0 mm, average pore size [dp] of 5.30 microns) at an average passing rate of 350 cc/ten minutes to re-emulsify whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 3.

Comparative Example 2 (W/O Type Emulsion)

(Preparation of Oily Phase) Soybean oil (50 parts) was heated at 60°C and, under stirring with a homo-mixer at 3,000 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (50 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by stirring with a homo-mixer at 3,000 rpm for ten minutes whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 3.

Example 3 (W/O Type Emulsion)

(Preparation of Oily Phase) Palm oil (mp: 34.7°C) (60 parts) was heated at 70°C and, under stirring with a homo-mixer at 5,000 rpm, 3.0% of polyglycerol condensed rinicoleate (SY Glyster CR-310; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 60°C.

(Preparation of Aqueous Phase) Water (40 parts) was heated at 70°C and, under stirring with a chemistirrer at 3,000 rpm, 0.5% of sucrose fatty acid ester of HLB = 16 (DK Ester F-160; manufactured by Daiichi Kogyo Seiyaku) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 60°C.

(Preparation of Preliminarily Emulsified W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by stirring with a homo-mixer at 5,000 rpm for ten minutes. After completion of the stirring, the mixture was allowed to stand at 60°C for ten minutes to deaerate.

(Preparation of W/O Type Emulsion) The preliminarily emulsified W/O type emulsion prepared as above was passed through a porous membrane (inner diameter of 9.0 mm; outer diameter of 10.0 mm, length of 100.0 mm, average pore size [dp] of 0.53 micron; previously warmed at 60°C by a jacket) at an average passing rate of 350 cc/30 minutes to re-emulsify whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 4.

Comparative Example 3 (W/O Type Emulsion)

(Preparation of Oily Phase) Palm oil (mp: 34.7°C) (60 parts) was heated at 70°C and, under stirring with a homo-mixer at 5,000 rpm, 3.0% of polyglycerol condensed rinicoleate (SY Glyster CR-310; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 60°C.

(Preparation of Aqueous Phase) Water (40 parts) was heated at 70°C and,

under stirring with a chemistirrer at 3,000 rpm, 0.5% of sucrose fatty acid ester of HLB = 16 (DK Ester F-160; manufactured by Daiichi Kogyo Seiyaku) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 60°C.

(Preparation W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by stirring with a homo-mixer at 5,000 rpm for ten minutes. After completion of the stirring, the mixture was allowed to stand at 60°C for ten minutes to deaerate whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 4.

Example 4 (O/W Type Emulsion)

(Preparation of Oily Phase) Rapeseed oil (30 parts) was heated at 60°C and, under stirring with a homo-mixer at 3,000 rpm, 1.0% of polyglycerol condensed rinicoleate (SY Glyster CR-310; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 11 (SY Glyster MO-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 3,000 rpm for ten minutes. After completion of the stirring, the mixture was deaerated *in vacuo* for ten minutes.

(Preparation of O/W Type Emulsion) The preliminarily emulsified O/W type emulsion prepared as above was passed through a porous membrane (inner diameter of 9.0 mm; outer diameter of 10.0 mm, length of 100.0 mm,

average pore size [dp] of 1.08 microns) at an average passing rate of 300 cc/20 minutes to re-emulsify whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 5.

Comparative Example 4 (O/W Type Emulsion)

(Preparation of Oily Phase) Rapeseed oil (30 parts) was heated at 60°C and, under stirring with a homo-mixer at 3,000 rpm, 1.0% of polyglycerol condensed rinicoleate (SY Glyster CR-310; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 11 (SY Glyster MO-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 3,000 rpm for ten minutes. After completion of the stirring, the mixture was deaerated *in vacuo* for ten minutes whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 5.

Example 5 (W/O Type Emulsion)

(Preparation of Oily Phase) Fractionated palm oil U (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of lecithin (manufactured by Ajinomoto) and 0.5% of glycerol fatty acid ester (Emulgy MS; manufactured by Riken Vitamin) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of polyglycerol fatty acid ester of HLB = 11 (SY Glyster TS-750; manufactured by Sakamoto Yakuhin Kogyo) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by stirring with a homo-mixer at 3,000 rpm for ten minutes. After completion of the stirring, the mixture was deaerated *in vacuo* for ten minutes.

(Preparation of W/O Type Emulsion) The preliminarily emulsified W/O type emulsion prepared as above was passed through a porous membrane (inner diameter of 9.0 mm; outer diameter of 10.0 mm, length of 100.0 mm, average pore size [dp] of 4.20 microns) at an average passing rate of 300 cc/30 minutes to re-emulsify whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 6.

Comparative Example 5 (W/O Type Emulsion)

(Preparation of Oily Phase) Fractionated palm oil U (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of lecithin (manufactured by Ajinomoto) and 0.5% of glycerol fatty acid ester (Emulgy MS; manufactured by Riken Vitamin) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of polyglycerol fatty acid ester of HLB = 11 (SY Glyster TS-750; manufactured by Sakamoto Yakuhin Kogyo) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of W/O Type Emulsion) The above-prepared aqueous phase part was gradually added to the above-prepared oily phase part followed by

stirring with a homo-mixer at 3,000 rpm for ten minutes. After completion of the stirring, the mixture was deaerated *in vacuo* for ten minutes whereupon a W/O type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 1, the emulsifying stability is shown in Fig. 1, and the particle size distribution is shown in Fig. 6.

Table 1 (Result of Average Particle Size Measurements)

	Diameter of PM (microns) (A)	APS of PEE (microns) (B)	APS of Emulsion (microns) (C)	B/A	C/A
Example 1	2.70	19.6	5.5	7.26	2.04
Comp.Ex.1			19.6		
Example 2	5.30	11.8	10.2	2.23	1.92
Comp.Ex.2			11.8		
Example 3	0.53	6.0	1.2	11.32	2.26
Comp.Ex.3			6.0		
Example 4	1.08	9.0	2.9	8.33	2.69
Comp.Ex.4			9.0		
Example 5	4.20	25.5	8.9	6.07	2.12
Comp.Ex.5			25.5		

PM: Porous membrane
APS: Average Particle Size
PEE: Preliminarily Emulsified Emulsion

Example 6.

(Preparation of Oily Phase) Corn oil (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the

oily phase 20°C.

(Preparation of Preliminarily Emulsified O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 3,000 rpm for five minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate.

(Preparation of O/W Type Emulsion) The preliminarily emulsified O/W type emulsion prepared as above was passed through a membrane filter (outer diameter of 47.0 mm, thickness of 35.0 microns, average pore size [dp] of 0.5 micron) of a porous membrane type (hydrophilic PTFE type membrane filter; manufactured by Toyo Filter Paper) under a pressure of 1.0 kg/cm^2 at an average passing rate of 500 cc/3 hours whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 2, the emulsifying stability is shown in Fig. 7, and the particle size distribution is shown in Fig 8.

Comparative Example 6.

(Preparation of Oily Phase) Corn oil (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol condensed rinicoleate (SY Glyster CR-500; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 3,000 rpm for five minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 2, the

emulsifying stability is shown in Fig. 7, and the particle size distribution is shown in Fig 8.

Example 7.

(Preparation of Oily Phase) Corn oil (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of lecithin (manufactured by Ajinomoto) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of Preliminarily Emulsified O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 5,000 rpm for ten minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate.

(Preparation of O/W Type Emulsion) The preliminarily emulsified O/W type emulsion prepared as above was passed through a membrane filter (outer diameter of 47.0 mm, thickness of 35.0 microns, average pore size [dp] of 1.0 micron) of a porous membrane type (hydrophilic PTFE type membrane filter; manufactured by Toyo Filter Paper) under a pressure of 1.5 kg/cm^2 at an average passing rate of 500 cc/2 hours whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 2, the emulsifying stability is shown in Fig. 7, and the particle size distribution is shown in Fig 9.

Comparative Example 7.

(Preparation of Oily Phase) Corn oil (30 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 1.0% of lecithin (manufactured by Ajinomoto) was added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the

temperature of the oily phase 20°C.

(Preparation of Aqueous Phase) Water (70 parts) was heated at 60°C and, under stirring with a chemistirrer at 620 rpm, 2.0% of polyglycerol fatty acid ester of HLB = 15 (SY Glyster ML-750; manufactured by Sakamoto Yakuhin Kogyo) were added thereto and dissolved therein. After completion of dissolution, temperature was adjusted so as to make the temperature of the oily phase 20°C.

(Preparation of O/W Type Emulsion) The above-prepared oily phase part was gradually added to the above-prepared aqueous phase part followed by stirring with a homo-mixer at 5,000 rpm for ten minutes. After completion of the stirring, the mixture was allowed to stand for ten minutes to deaerate whereupon an O/W type emulsion was prepared. Results of measurement of particle sizes of the resulting emulsion is shown in Table 2, the emulsifying stability is shown in Fig. 7, and the particle size distribution is shown in Fig 9.

Table 2 (Result of Average Particle Size Measurements)

	Diameter of PM (microns) (A)	APS of PEE (microns) (B)	APS of Emulsion (microns) (C)	B/A	C/A
Example 6	0.5	9.8	1.4	19.6	2.8
Comp.Ex.6			9.8		
Example 7	1.0	9.4	2.4	9.4	2.4
Comp.Ex.7			9.4		

PM: Porous membrane
APS: Average Particle Size
PEE: Preliminarily Emulsified Emulsion

It is apparent from the above-mentioned results for emulsifying stability and average particles size for the emulsions prepared in Examples 1-7 and Comparative Examples 1-7 that the emulsions of Example 1-7 which were prepared by re-emulsification of the preliminarily emulsified emulsion by passing through a porous membrane showed significantly improved emulsifying stability, smaller particle size of the emulsion and narrower particle size distribution width as compared with the emulsions of Comparative Examples 1-7 where no re-emulsification using a porous membrane was not carried out.

[Advantage of the Invention]

As mentioned hereinabove, it is now possible in accordance with the present invention to offer an emulsified fat/oil composition having uniform emulsion particle size without a sudden rising of viscosity. When the pore size of the porous membrane used is appropriately selected, it is possible to control an average particle size to 1- to 3-fold of the said pore size. Consequently, separation of aqueous and oily phases and hygienic problems caused by the particle size distribution can be solved whereby it is now possible to offer an emulsion having physical property, feel on eating and taste which have not been available up to now.

Brief Explanation of the Drawings:

Fig. 1 is a graph showing the result of measurement of emulsifying stability of the emulsions. Fig. 2 is a graph showing the result of measurement of particle size distribution of the emulsions. Fig. 3 is a graph showing the result of measurement of particle size distribution of the emulsions. Fig. 4 is a graph showing the result of measurement of particle size distribution of the emulsions. Fig. 5 is a graph showing the result of measurement of particle size distribution of the emulsions. Fig. 6 is a graph showing the result of measurement of particle size distribution of the emulsions. Fig. 7 is a graph showing the result of measurement of emulsifying stability of the emulsions. Fig. 8 is a graph showing the result of measurement of emulsifying stability of the emulsions. Fig. 9 is a graph showing the result of measurement of emulsifying stability of the emulsions.

粘度上昇等を伴うことなく、エマルション粒子径が均一化された乳化油脂組成物を提供することが出来る。また、用いる多孔質膜の孔径を適宜選択することで、膜孔径の1～3倍の平均粒子径にコントロールすることができる。かくして、粒子径の分布に起因する水相および油相の分離、衛生上の問題を解消することが出来、従来にない物性、食感、風味を有したエマルションが得られる。

【図面の簡単な説明】

【図1】 エマルションの乳化安定性測定結果を示すグラフ

【図2】 エマルションの粒径分布測定結果を示すグラフ

【図3】 エマルションの粒径分布測定結果を示すグラフ

【図4】 エマルションの粒径分布測定結果を示すグラフ

【図5】 エマルションの粒径分布測定結果を示すグラフ

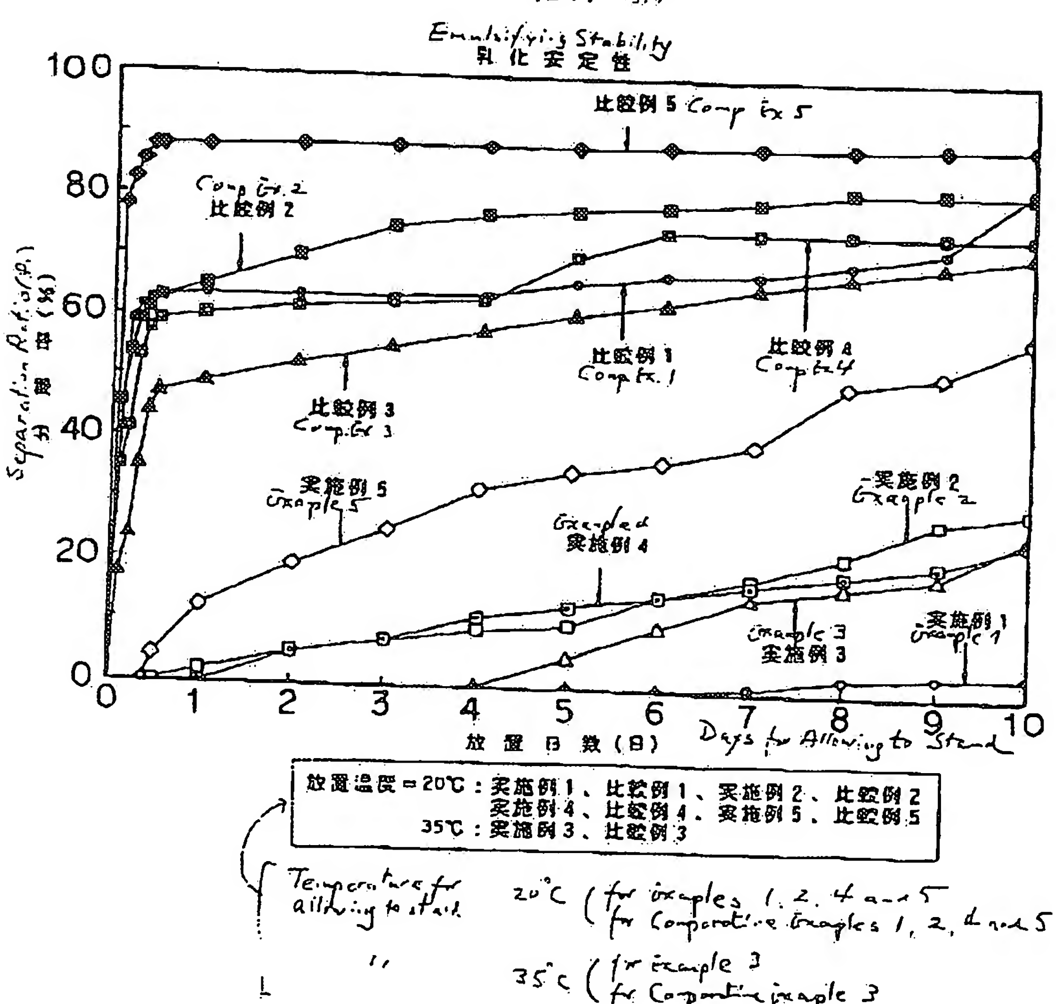
【図6】 エマルションの粒径分布測定結果を示すグラフ

【図7】 エマルションの乳化安定性測定結果を示すグラフ

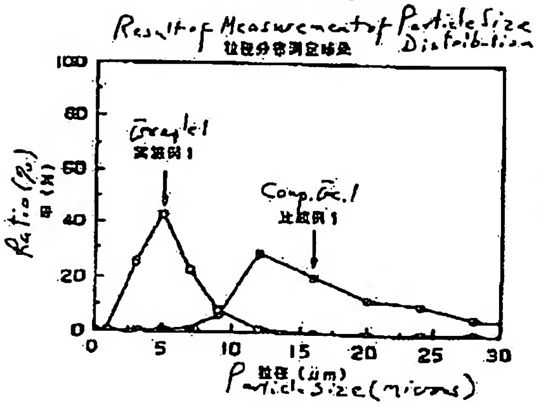
【図8】 エマルションの粒径分布測定結果を示すグラフ

【図9】 エマルションの粒径分布測定結果を示すグラフ

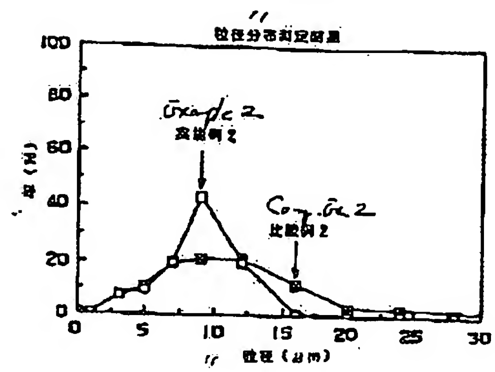
【図1】 Fig. 1



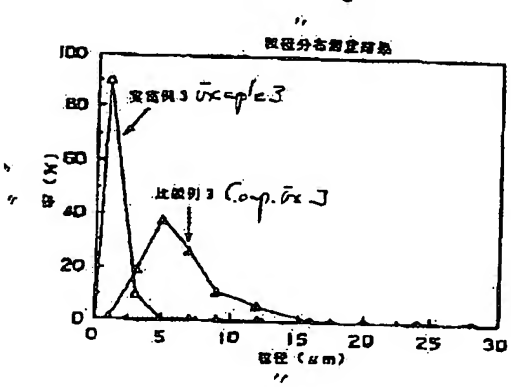
【図2】Fig 2



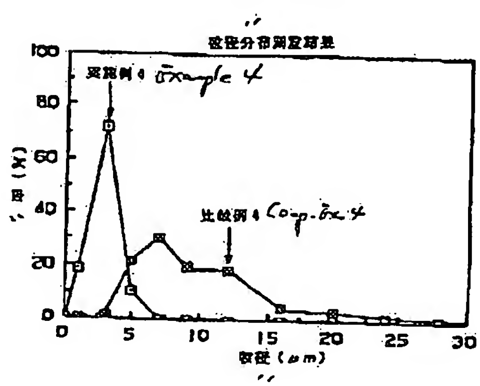
【図3】Fig 3



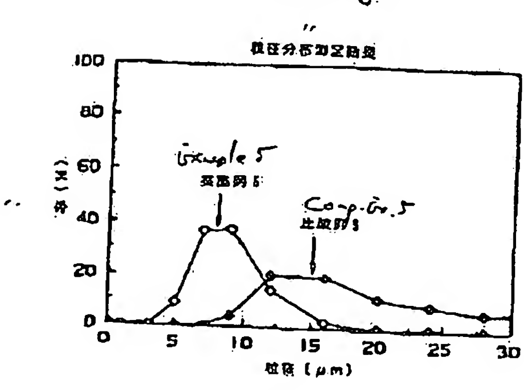
【図4】Fig. 4



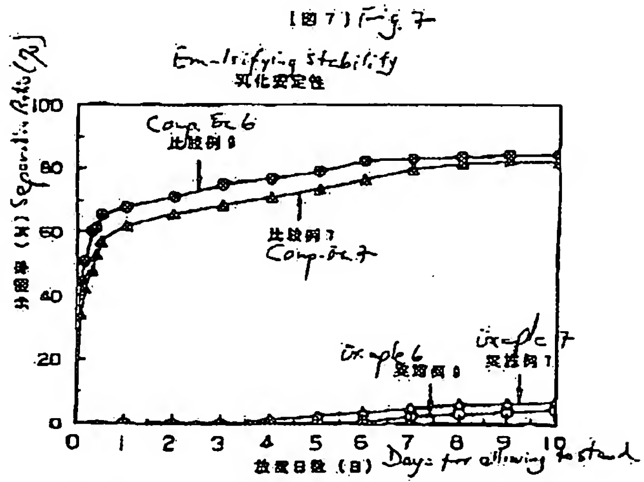
【図5】Fig. 5



【図6】Fig. 6



【図7】Fig. 7



放置温度＝２０℃：実施例６、比較例６、実施例７、比較例７

Temperature = 20°C for Examples 6 and 7 and Comparative Examples 6 and 7